COMPARISON BETWEEN ISO AND ASTM METHODS FOR POLYPROPYLENE INJECTION MOLDING AND MECHANICAL TESTING

by

Henry J. Sokol

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ABSTRACT

Two primary testing protocols, ASTM and ISO, are commonly used to standardize the injection molding and analytical testing of polypropylene resin samples. These two methods are similar in many ways, but differences between the two can have an effect on the reported properties of molded parts. An understanding of the variation between reported results from each method for many resin types is important to be able to demonstrate and verify plastic product performance. In this investigation, a wide selection of polypropylene samples was chosen to be injection molded and analyzed using multiple testing mechanisms. Several injection conditions were varied to gain understanding of the effects of temperature, pressure, and speed on the properties of molded samples. A direct comparison between data obtained through ISO and ASTM methods was also performed. The experimental results were further verified through the use of computer simulation data. Multiple simulations were done to gather information about parameters during the injection molding process that could not be measured experimentally. With these results, correlations between ISO and ASTM reported properties were established for flexural, tensile, impact resistance, and optical tests. It was found that the mold temperature and test bar dimensions can have a significant impact on the final properties of a molded part. The comparison data showed that the flexural modulus and tensile strength at yield of polypropylene samples is very similar when measured using ISO or ASTM procedure. However, for several polypropylene resin types the impact resistance was found to vary when different injection and testing methods were used. With the findings from this report, it is possible to compare ISO and ASTM reported mechanical performance values for the majority of polypropylene resins.

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INTRODUCTION

Standardized polymer testing is a critical aspect of the plastics industry. The ability to accurately demonstrate and verify the performance and properties of products on a consistent and reliable basis is very important. To accomplish this, polymer samples are injection molded and then tested to determine a variety of mechanical, thermodynamic, and optical properties. This procedure is usually carried out in accordance with one of two main testing protocols, American Society of Testing Materials (ASTM) or International Standards Organization (ISO). ASTM standards are primarily used in North America while ISO standards are used primarily throughout Europe and Asia. While ASTM and ISO injection molding and testing procedures exhibit many similarities, there are some significant differences between the two methods that can lead to different properties in molded parts. An understanding of these differences is important to be able to compare reported product properties between ISO and ASTM methods. This investigation uses data obtained under both standards in order to establish correlations between the two and to explain the effects of various parameters in each method on reported polypropylene properties.

The most significant differences between the ASTM and ISO methods are the dimensions of the molded parts and the injection molding conditions. The parameters used during molding can have a significant effect on the morphology and crystal structure of a molded polypropylene specimen, leading to differences in mechanical properties when tested. During the molding process, polymer resin is heated to the melt temperature, and injected under pressure into a temperature controlled mold. The pressure is continuously applied during the packing phase to fill the mold. Then the polymer melt is allowed to cool during the cooling phase, until the solidified part is ejected from the mold. After conditioning the specimen

in a controlled atmosphere for a duration specified by ASTM or ISO, it is ready to be tested. The primary tests used to determine the mechanical properties of a polypropylene sample are flexural, tensile, and impact testing.

The morphological structure of a polymer has a significant impact on its mechanical and optical properties. In an injection molded polypropylene part, there are two distinct internal structural layers that show different levels of orientation and crystallization.[1,2] These include a highly oriented outer layer and a crystalline core layer. The outer layer is made up of a thin skin formed when the molten polymer contacts the mold wall, a transition layer formed by the flow front of the polymer melt, and a shear layer of highly oriented chains. The core layer is formed at a lower cooling rate that allows for more chain relaxation and the formation of spherulitic crystalline structures. These layers contain different structural components and attributes such as orientation, crystallinity, amount of crystal phase and lamellar thickness. The thickness of these layers can be influenced by the polymer molecular weight , molecular weight distribution, and ethylene content as well as the parameters used in the molding process and can have a significant effect on the mechanical properties of the polymer sample.[3] It is important to understand how molding conditions under ISO and ASTM standards influence the thickness, orientation, and crystallinity of each layer and the effect that has on the final mechanical properties of the molded polymer sample. The specific conditions that this investigation focuses on include mold and melt temperature, cycle time, and injection and packing pressure, as these are the primary parameters that differ between ASTM and ISO injection molding.

During injection molding, the cooling rate of the molten polymer sample has a significant effect on its structural formation and mechanical properties. [3] This rate is heavily influenced by the interaction between the melt temperature of the polymer resin before injection

and the temperature of the mold into which it is injected. For ASTM and ISO standards, the specified melt temperature is based on the melt flow rate (MFR) of the selected resin. For many polymer samples, both molding protocols call for the same melt temperature, however there are certain MFR ranges in which the specified temperature can differ by as much as 20°C. The melt temperature can have a significant effect on the thickness of the skin layer in a molded part.[2] Lower melt temperatures typically produce parts with a thicker skin layer, which allows for a smaller spherulitic core layer. It has been reported that lower melt temperatures can contribute to a decrease in flexural strength and flexural modulus and an increase in impact resistance properties.[4,5] This has also been shown in injection molded samples of polypropylene composites that showed signs of thermal degradation at high melt temperatures.[6] Along with the polymer melt temperature, the mold temperature plays an important part in the cooling of the molded sample. Injection molding mold temperatures differ between 40°C for ISO molding and 60°C for ASTM. It has been shown that this temperature affects the cooling rate of the polymer melt which can produce differences in the skin and core layering and the crystal structure.[3] Higher mold temperatures contribute to slower cooling, which leads to molded parts with a smaller skin layer and a thicker core. This can have an impact on a sample's mechanical properties as higher mold temperatures have been found to produce parts with a higher flexural modulus.[4] The same findings have been reported in micro-injection molded PP.[7] In addition, faster cooling rates contribute to less crystal growth and smaller crystal size within a polymer which can affect the final mechanical properties of a molded part.[8, 9] This has also been shown in samples containing nucleating agents where it has been reported that faster cooling decreased the crystal fraction for nucleated samples.[10] With this previous research in mind, it is important to consider the differences in melt temperature and mold temperature between ISO and ASTM standards.

Several other parameters during the injection and packing stages of the molding process can also influence the mechanical properties of a sample. ASTM and ISO molding protocols specify different packing times and usually must employ different packing and injection pressures. ASTM uses a packing time of 15 seconds where ISO uses 40 seconds, and neither method specifies a strict pressure value. ISO and ASTM standards also use different injection times for injection molding. ASTM injection time is constant at 5 seconds, while ISO injection times vary between 1 and 2 seconds depending on the volume and number of cavities in the mold being used. The shorter injection times specified by ISO molding mean that higher pressures are commonly used for injection and packing in order to produce a full part in a shorter time. These differing conditions can affect the shear rates on the polymer melt during molding and can contribute to differences in the final part. It has previously been found that higher packing pressure has produced parts with lower tensile yield strength and higher impact strength results.[4] In addition, higher packing pressure has been shown to reduce flexural modulus for molded samples.[5] These results are likely due to the higher shear during molding of parts at high pressures. This leads to an increase in the thickness of the oriented layers within the molded part, which has been shown to increase the impact strength of certain polypropylene samples.[2,11] Slower injection speeds have also been shown to have an impact on the formation of the core and skin layers in molded specimens.[1,3] The thickness of the various layers has a significant effect on the crystallinity and the modulus of the molded sample. It has been found that slower injection speeds produced samples with higher flex and tensile modulus and lower impact resistance.[4] The pressure and time of the injection and packing phases of the molding process are important parameters that can contribute to the final mechanical properties of a polymer sample. It is critical to consider the differences in these parameters between the ASTM and ISO methods and the effects this may have on mechanical properties test results.

For this investigation, samples were chosen to represent a wide variety of polypropylene resins. Polypropylene homo-polymers (HPP) as well as impact co-polymers (ICP) and random co-polymers (RCP) were used. Among the selected resins a wide range of MFRs is represented. MFR is a measure of how easily a melted polymer flows through a capillary die and is reported in grams of material per ten minutes.[12] MFR is highly dependent on the molecular weight, molecular weight distribution, and viscosity of a polymer sample. For the HPP samples used in the study, a wide range of xylene soluble content (%XS) samples were selected. %XS can be an indication of the crystallinity of the polymer sample as the amorphous phase is soluble while the crystalline phase is not.[13] There is an approximately inversely proportional correlation between %XS and crystallinity. Along with the HPP samples, a range of ICPs was also used in this investigation. ICPs are a class of polymer resin that is made up of two parts: HPP and poly(ethylene-co-propylene) in the rubber phase.[14,15,16] This poly(ethylene-co-propylene) is a polymer in which blocks of polypropylene and polyethylene are attached to form the molecular chains. ICPs are generally used to obtain better toughness than standard polypropylene samples. The addition of the rubber phase ethylene propylene copolymer is usually done in order to increase impact strength and make a polymer sample more ductile.[17] Multiple ICPs were used to represent a wide range of rubber fraction content (Fc). Fc is a measure of the fraction of the polymer made up of ethylene-propylene rubber blocks and has a significant impact on the properties of the polymer. In addition, two RCP samples were molded and tested in this study. RCPs contain both propylene and ethylene randomly structured throughout the polymer chain.[14] With the wide range of resins considered in this investigation, both nucleated and un-nucleated grades were used. This was done as it has been shown that the presence of nucleating agents has an effect on the crystallization of injection molded polymer specimens and therefore has an impact on mechanical properties.[18] Previous studies have found that the

presence of nucleating agents can increase the flexural modulus of polymer samples. By considering a wide range of polypropylene resins, it was possible to obtain correlations between ISO and ASTM results for the majority of industrial polypropylene resin grades.

EXPERIMENTAL

Materials

For this investigation, a total of 25 resin samples were used in order to represent the wide range of polypropylene types commercially available. The samples tested included 8 HPPs, 11 ICPs, and 2 RCPs (Table 1-3). For each class of polypropylene, resins were chosen with a range of melt flow rates to provide a representative sample of all products. In addition, resins with differing rubber fraction content (Fc), ethylene content and %XS were used. The HPP samples selected represent a range of MFR's from 0.1 to 47 g/10min and a %XS range from 1.0 to 5.5. The ICP samples ranged from 5 to 75 g/10min MFR and from 12 to 32 Fc. The grades chosen include both nucleated and un-nucleated samples.

HPP Resins	MFR	%XS
HPP1	3.5	5.5
HPP2	1.6	1.0
HPP3	8	1.0
HPP4	47	2.6
HPP5	17	2.6
HPP6	3	2.8
HPP7	0.3	3.7
HPP8	0.1	3.0

ICP Resins	MFR	Fc
ICP1	6	15
ICP2	20	32
ICP3	12	22
ICP5	75	22
ICP6	55	22
ICP7	16	32
ICP8	20	33
ICP9	6	12
ICP10	6	24
ICP11	5	24

Table 1: Polypropylene resin sample selection for HPP resins (left), and ICP resins (right).

Procedure

Three sets of experiments were done to investigate the impact of differences between ISO and ASTM methods for polypropylene injection molding and analytical testing on the final properties of molded parts. In the first experiment, the parameters and conditions used during

the injection molding process and their effect on specimen properties were investigated. Two samples, an ICP and a HPP resin, were molded with four different sets of conditions. The HPP sample selected had a MFR of 3.2 g/10min. and was 5.5% XS. The ICP sample had a MFR of 6 g/10min. and a rubber fraction content of 15%. The molding parameters used were selected to combine elements of ASTM and ISO procedure in order to investigate differences between the two methods. The primary parameters investigated were mold temperature and cycle time. The molding conditions were: 1) standards defined by ASTM specification, 2) ASTM procedure with a mold temperature of 40°C as specified by ISO molding procedures, 3) ASTM procedure with a cycle time of 60 seconds following ISO procedure, and 4) ASTM procedure with a 40°C mold temperature and 60 second cycle time. For each set of conditions, specimens were molded and tested for flexural modulus, tensile strength at yield and notched Izod impact resistance.

After molding and conditioning according to ASTM procedure, the samples were tested to measure flexural, tensile and impact resistant properties. Tensile testing was performed according to ASTM D638 and ISO 527 standards. Izod impact testing was performed using ISO 180 and ASTM D4101 specifications. Flexural testing was done according to ISO 178 and ASTM D790A methods.

In the second experiment, a direct comparison between ISO and ASTM testing was conducted using eight HPP, eleven ICP, and two RCP resins. For these tests, samples were injection molded and tested according to either ASTM or ISO specifications. Five ICP samples were also Izod and Charpy impact tested at 0°C and -30°C. For these specimens, the test bars were conditioned according to ASTM or ISO specification and placed in a freezer approximately six hours prior to testing. The specimens where then quickly transferred from the freezer to the testing apparatus to ensure testing at the specified cold temperature. Room temperature

samples were conditioned and tested according to ASTM or ISO specifications. ASTM Izod results were converted from the usual J/m into kJ/m^2 for comparison with ISO samples.

The third experiment investigated the impact of injection molding conditions on the optical properties of a sample. Two clarified ICP resins where molded into step-chips for optical testing. Eight different sets of molding conditions were used for each resin (Table 2). The melt temperature, mold temperature, injection pressure, and injection velocity were varied to investigate impact on optical properties. Each sample was then tested for haze, clarity, and gloss and the results from each set of molding conditions were compared.

Conditions	Melt Temp (°C)	Mold Temp (°C)	Injection Pressure (bar)	Injection Velocity (cm^3/s)
1	190	20	800	60
2	190	45	400	32
3	210	20	400	32
4	210	45	400	32
5	210	45	400	60
6	210	60	400	32
7	230	60	400	32
8	230	45	400	32

Table 2: Injection molding conditions used for optical testing.

Modeling

In order to gain a better understanding of conditions during the injection molding process, computer simulations were done to investigate the cooling rates, crystallinity and shear rates inside of injection molded bars. Simulations were set to mimic ISO and ASTM conditions within injection molded tensile, flex, and Izod bars. The first modeling program simulated the relative crystallinity and flow induced stress profiles across an ASTM bar injected into different

temperature molds. Further simulations were also done on the temperature profile, and relative crystallinity through the thickness of the cross section of both ASTM and ISO bars during the injection molding process. In addition, the relative crystallinity and volume shrinkage in step-chip molds for optical testing were simulated at various mold temperatures. These simulations were done to investigate the injection molding process through parameters that could not be measured experimentally with the available equipment.

RESULTS AND DISCUSSION

Injection Molding Study

Izod impact, tensile, and flexural testing was done as a measure of the mechanical properties of a HPP and an ICP resin sample under different molding conditions. These tests were used to gain understanding into the impact of molding parameters on the final properties of a molded part. Cycle time and mold temperature were found to have an impact on the Izod impact resistance in the HCP sample and mold temperature alone affected the impact resistance of the ICP sample (Figure 1). It was found that decreasing the mold temperature from 60°C to 40°C and increasing the injection molding cycle time from 45 seconds to 60 seconds contributed to an increase in impact resistance in the HPP resin. The HPP sample showed around a 50% increase in impact resistance when cycle time was changed to 60 seconds, and around a 30% increase when the mold temperature was lowered to 40°C. When both cycle time and mold temperature were changed, the impact resistance increased to its highest level at about 60% more than the ASTM method value. For the ICP resin, using a longer cycle time did not change the measured value for impact resistance, and a colder mold led to only around a 5% increase, remaining within the error range of the 60°C mold temperature sample. When both conditions were changed, the impact resistance was the same as when measured following ASTM protocol. The impact resistance of the more ductile ICP sample was not affected by molding parameters in the same way as the more brittle HPP. When testing this ICP resin, the polymer's impact properties were not influenced by the molding conditions used.

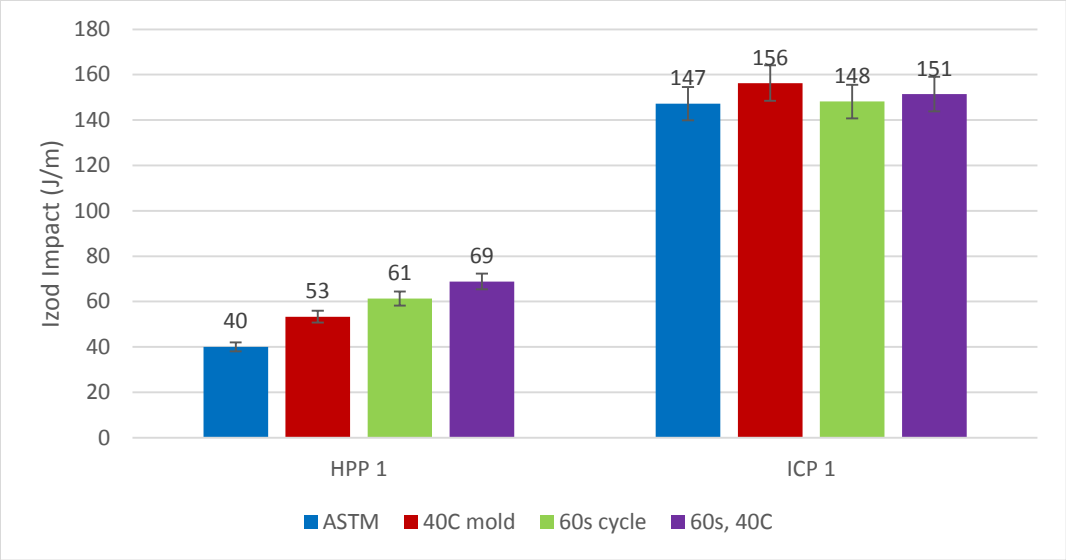

Figure 1: Izod impact resistance at 23°C of samples molded under varying conditions

The flexural modulus was also tested for the HPP and ICP sample using various molding conditions. The results show that for both the HPP and the ICP sample, increasing the cycle time from 45 seconds to 60 seconds had no meaningful effect on the modulus of the sample (Figure 2). However, lowering the mold temperature from 60°C to 40°C decreased the modulus very slightly by less than 5%. When both mold temperature and cycle time were changed, the modulus was consistent with the result obtained with a 40°C mold temperature. These trends were evident in both the HPP and ICP sample. All of the modulus values measured were within the error range of each other, indicating that while mold temperature may have an impact on the flexibility of a polymer resin, the reported modulus remains consistent.

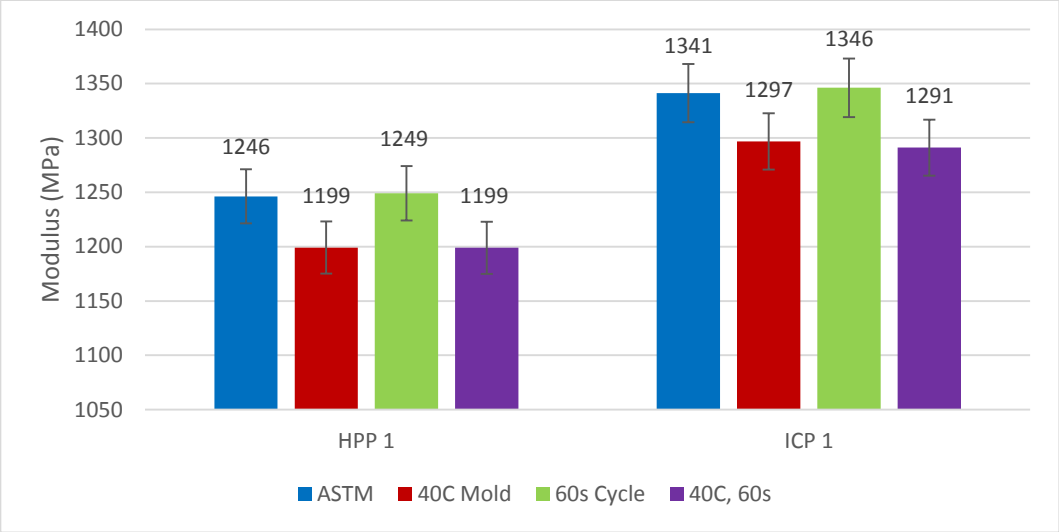

Figure 2: Flexural modulus at various molding conditions

The tensile strength at yield of the HPP and ICP samples with different molding conditions was also tested and reported (Figure 3). It was found that changing the mold temperature form 60°C to 40°C or the cycle time form 45 seconds to 60 seconds had no meaningful impact on the tensile strength of either the HPP or the ICP sample.

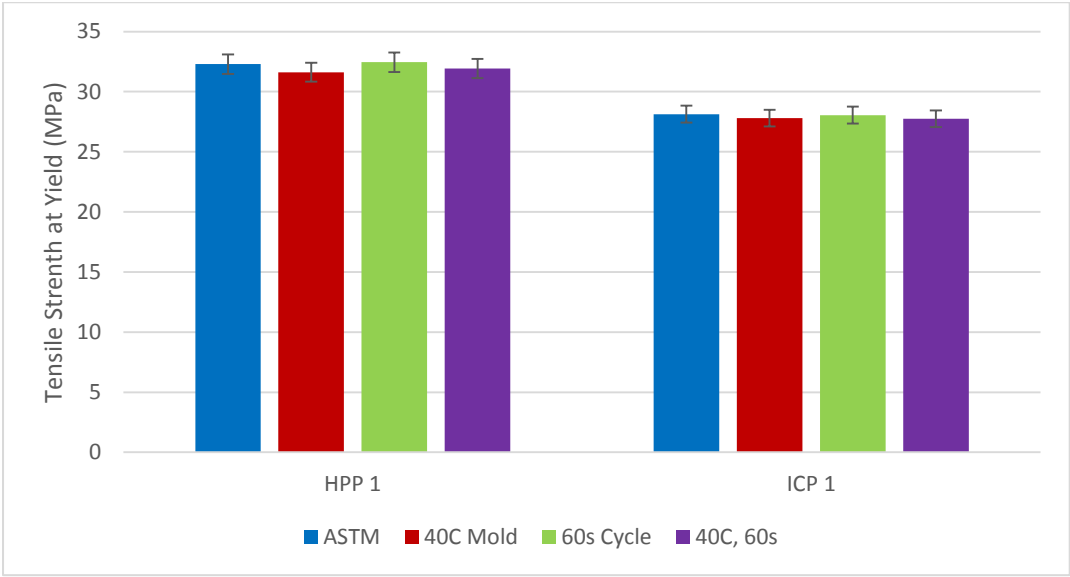

Figure 3: Tensile strength at yield with various molding conditions

Varying the mold temperature by 20°C and the cycle time by 15 seconds did not have a large influence on the mechanical properties of the molded parts. Decreasing mold temperature

produced a sample with an increased Izod impact resistance for the HPP and a very slightly decreased flexural modulus for both the HPP and ICP resin. This can be largely explained through the cooling rate of the polymer samples and the effect that has on the crystalline structure. The higher mold temperature of 60°C induced slower cooling in the sample than in samples injected into a 40°C mold. At a faster cooling rate, the crystallinity and spherulite size in the sample is limited, which can impact the mechanical properties of the sample.[19] In addition, a lower mold temperature has the effect of freezing the sample skin layer in a highly oriented state. As a result, we see samples injected into a lower temperature mold experience a faster cooling rate and show higher impact resistance and a slightly lower flexural modulus. This is as expected as a sample with lower overall crystallinity and smaller crystal spherulites is less brittle and stiff due to a larger portion of the polymer in the amorphous phase allowing more flexibility between spherulites. The ICP sample tested contains a rubber phase amorphous region with relatively high ductility so its impact resistance was not affected by cooling rate in the same way as the more brittle HPP resin.

The cycle time used for the injection process was only found to affect the Izod impact resistance of the HPP sample and not the ICP sample. Cycle time did not change the values recorded for tensile strength or flexural modulus. This is likely due to the faster injection and longer packing time in the 60 second cycle. With these parameters, slightly more polymer melt can be packed into the molded bar. This small amount had no noticeable impact on tensile strength, modulus or the high impact resistance of the ductile ICP sample. It did however strengthen and lead to an increase in the impact resistance of the more brittle HPP sample.

ASTM and ISO Testing Comparison

The second set of experiments done was a direct comparison between the ISO and ASTM methods for injection molding and mechanical properties testing. A wide range of HPP, ICP, and RCP samples were used for this experiment. The Izod impact resistance, flexural modulus, and tensile strength at yield of each sample was record using both ISO and ASTM procedure so that the two methods could be directly compared.

For the HPP samples tested, the Izod impact resistance results using ASTM and ISO were compared (Figure 4). It was found that the ISO procedure measured an impact resistance approximately 1.3 times higher than the result obtained following ASTM procedure for HPP samples. This correlation was found when units of kJ/m^2 are used and does not necessarily apply if J/m are used. Because of the differing dimensions of the samples, comparisons using units of energy per cross sectional area and energy per depth may not lead to the same conclusion.

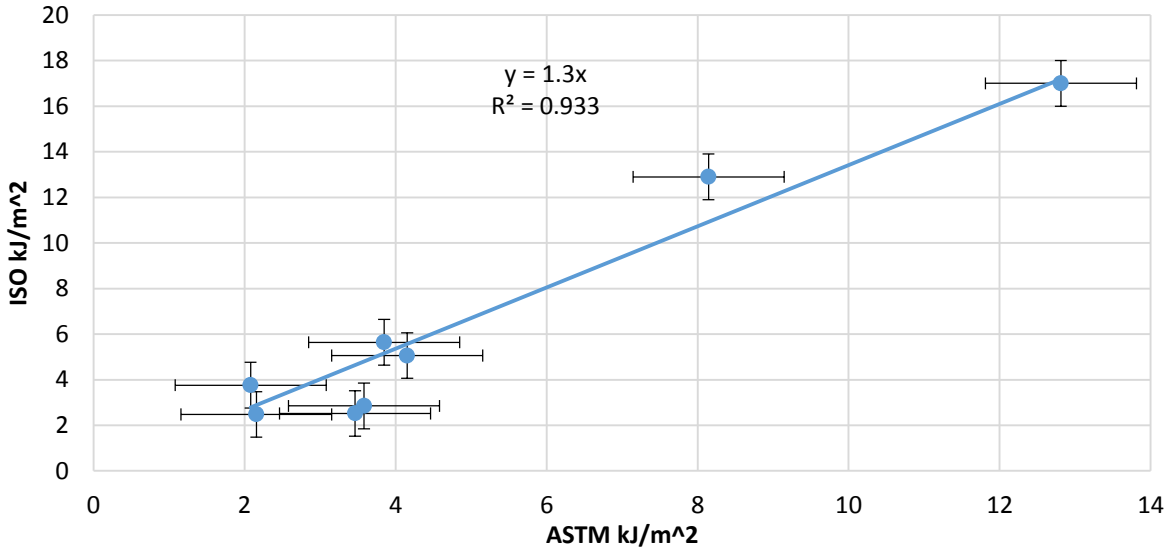

Figure 4: HPP Izod impact resistance comparison between ISO and ASTM

The ISO and ASTM Izod impact resistance results were plotted as a function of sample MFR and %XS (Figure 5). There was no correlation between the difference between ISO and

ASTM impact resistance results and sample %XS. The greatest difference between ISO and ASTM impact results was found for the samples with very low MFR (<1 g/10 min), with all other samples showing near equivalent ISO and ASTM impact resistance.

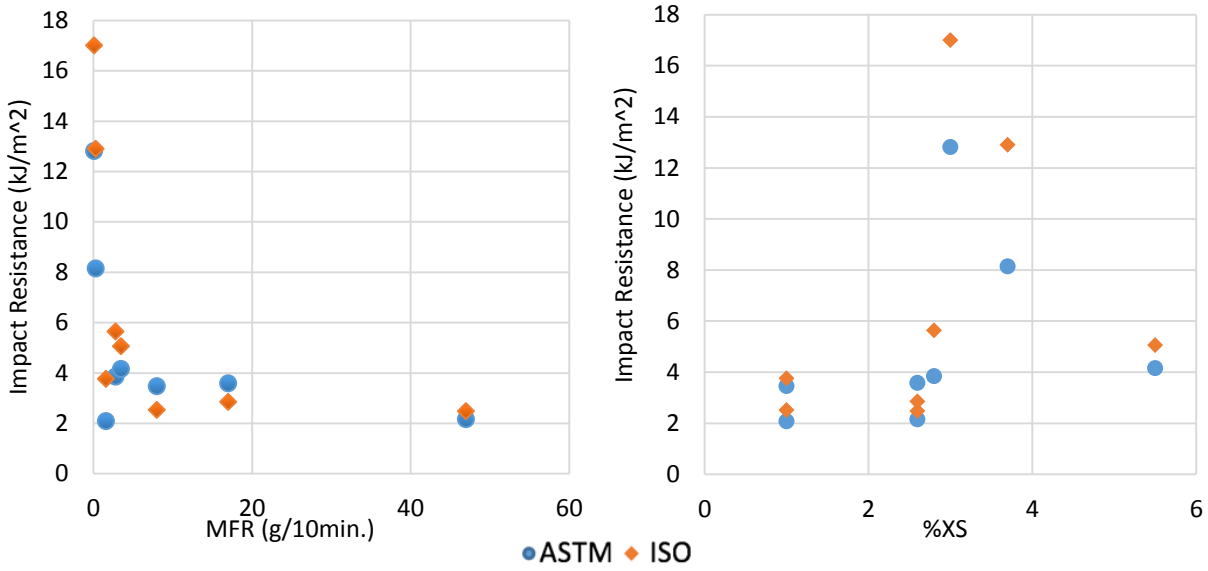

Figure 5: HPP Izod impact resistance comparison as a function of sample MFR (left) and sample %XS (right).

From the experiments on the effect of injection molding conditions, it is expected that the ISO method injection parameters would produce samples with a higher impact resistance. However, in HPP resins this was only found to be true in samples with low MFR with high measured impact resistance. Since there are no differences between the testing techniques for ISO and ASTM, the differences in the bar dimensions for each method must contribute to the measured Izod values. This is the reason for only negligible differences in impact resistance between ISO and ASTM except for at very low MFR. At low MFR, the injection molding conditions have a more pronounced effect on the structure of the sample. In these high molecular weight, long polymer chain samples, the colder mold temperature used for ISO molding likely has a greater contribution to decreased crystallinity and spherulite size than in other samples. As longer polymer chains with a high relaxation time take longer to form crystal

spherulites than higher MFR samples, the effects of cooling rate are more pronounced at low MFR. This leads to the measured difference in impact resistance between the two methods in the very low MFR HPP resins.

The HPP samples were also flex tested using both ASTM and ISO procedures (Figure 6). For this test the flexural modulus is calculated in a different manner when following ISO or ASTM protocol. The ISO modulus is calculated as a 0.05% strain to 0.25% strain chord modulus, and the ASTM modulus is a 1% strain secant modulus. In the majority of HPP samples there was found to be no meaningful difference between flexural modulus results obtained through each methods' procedures.

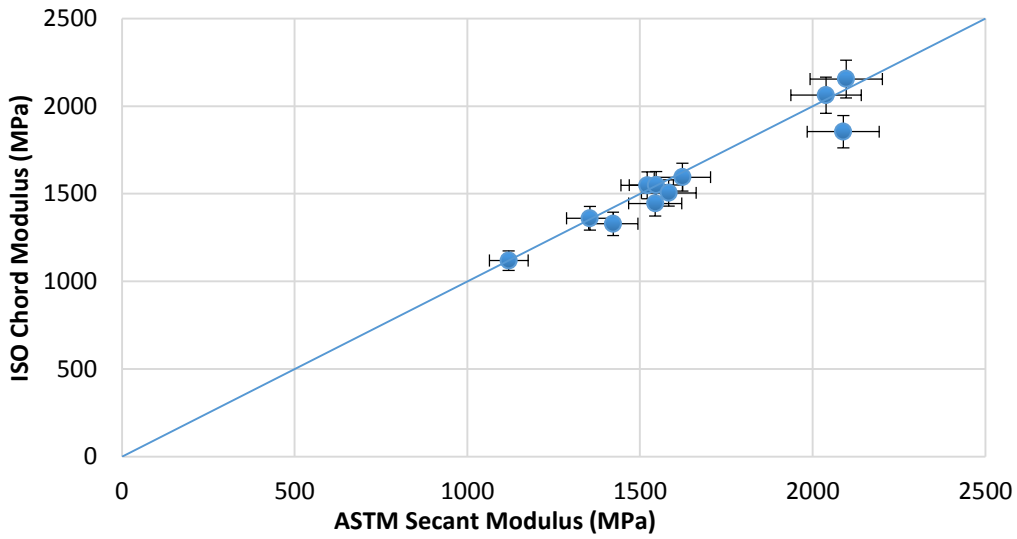

Figure 6: HPP flexural modulus comparison between ISO and ASTM methods

When plotted as a function of sample MFR or sample %XS, there is no evident correlation with the samples showing a slightly higher ASTM modulus than ISO modulus. HPP samples across the entire range of MFR and %XS show no significant difference in modulus between ISO and ASTM methods (Figure 7). The previous injection molding parameter experiments indicated that the colder mold temperature used for ISO molding may lead to a

slightly reduced flexural modulus in ISO method samples than in ASTM. However, this was found to not be the case as the modulus was not dependent on the molding and testing method used. The likely explanation for this is differences in bar dimensions and testing procedure. While the colder mold suggests a lower modulus with the ISO method, the thicker sample size still allowed for slow cooling and crystallite formation in the core region so the modulus is consistent. The net result was no noticeable difference in modulus between ISO and ASTM methods for HPP samples.

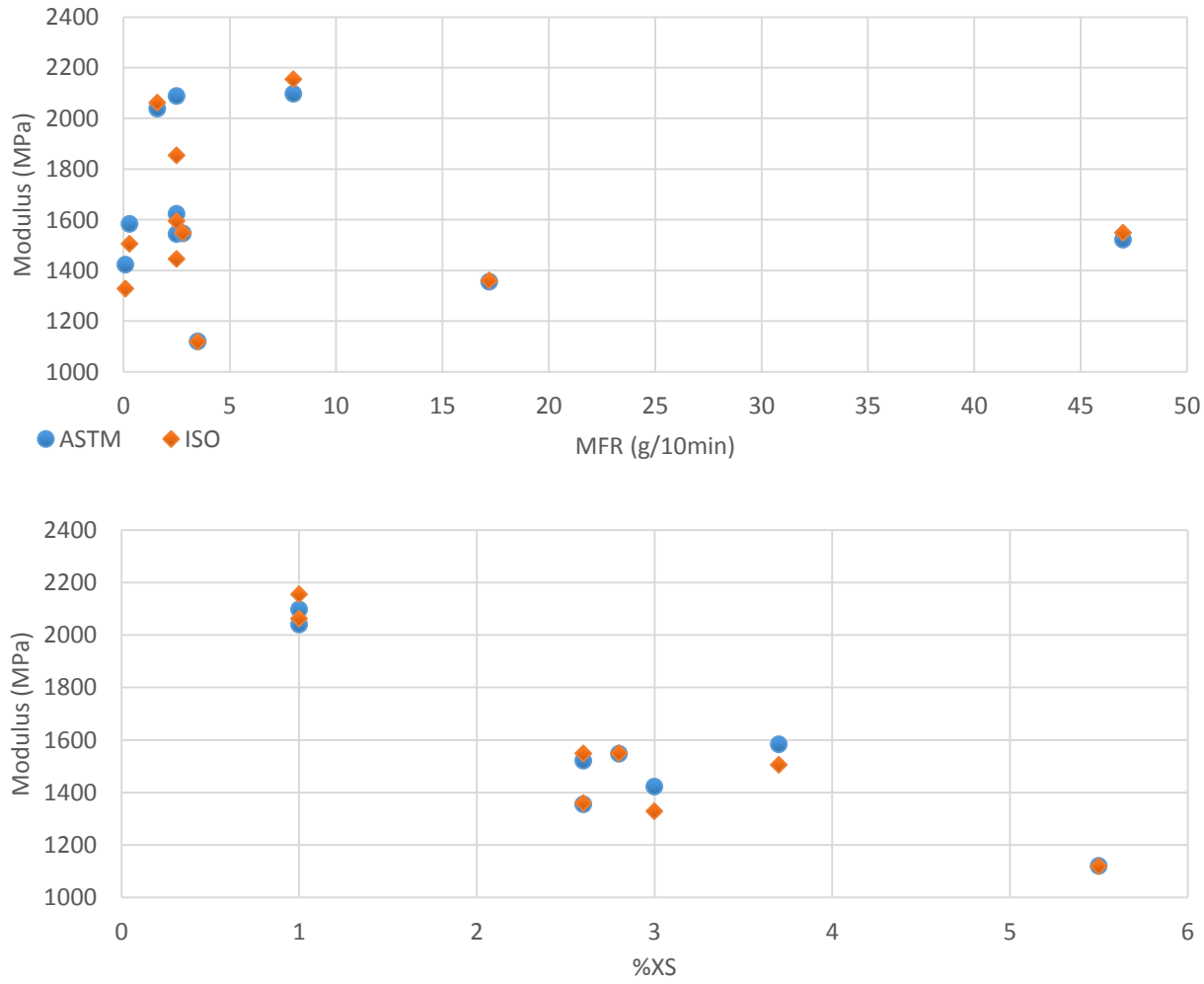

Figure 7: HPP flexural modulus as a function of MFR (top) and as a function of sample %XS (bottom).

Tensile strength at yield was also measured for the HPP samples used in this test. The majority of HPP samples reported a slightly higher tensile strength at yield with the ASTM

method than with the ISO method (Figure 8). ASTM results were around 1 MPa greater than ISO results for the majority of samples tested. When considering for a 5% error in the tensile strength measurements, there was no significant difference between results from ISO and ASTM methods.

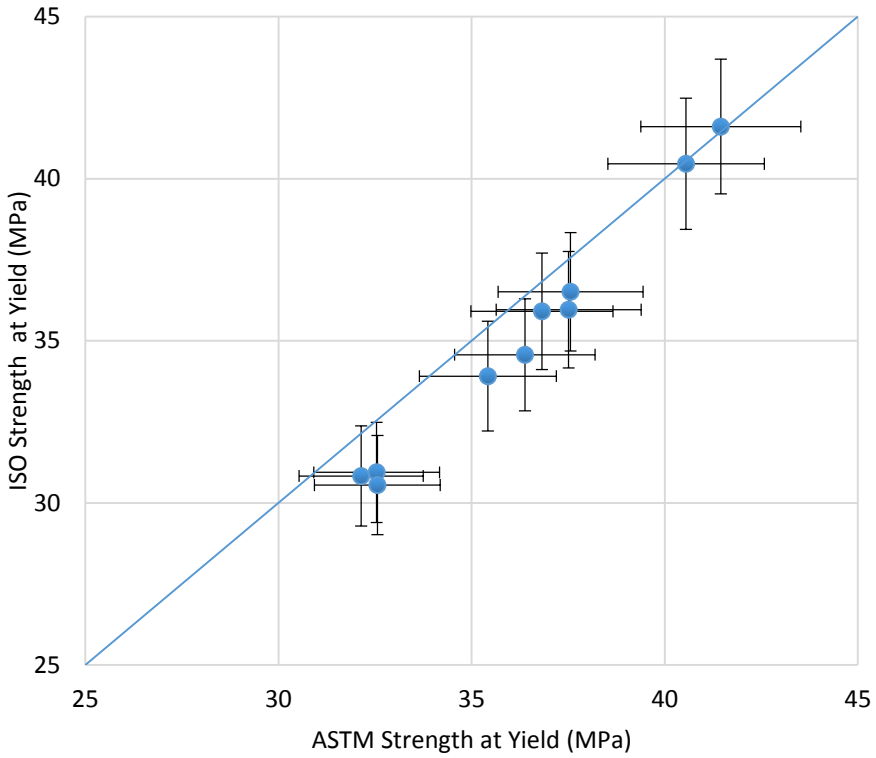

Figure 8: HPP tensile strength at yield ISO and ASTM comparison

The tensile strength at yield results were broken down as a function of sample MFR and sample %XS (Figure 9). HPP samples with very low %XS near 1% showed no difference between ISO and ASTM tensile strength at yield while the other HPP resins showed a very slight increase in strength using the ASTM method. Resin MFR was shown not to be a factor in the difference between tensile results from each method.

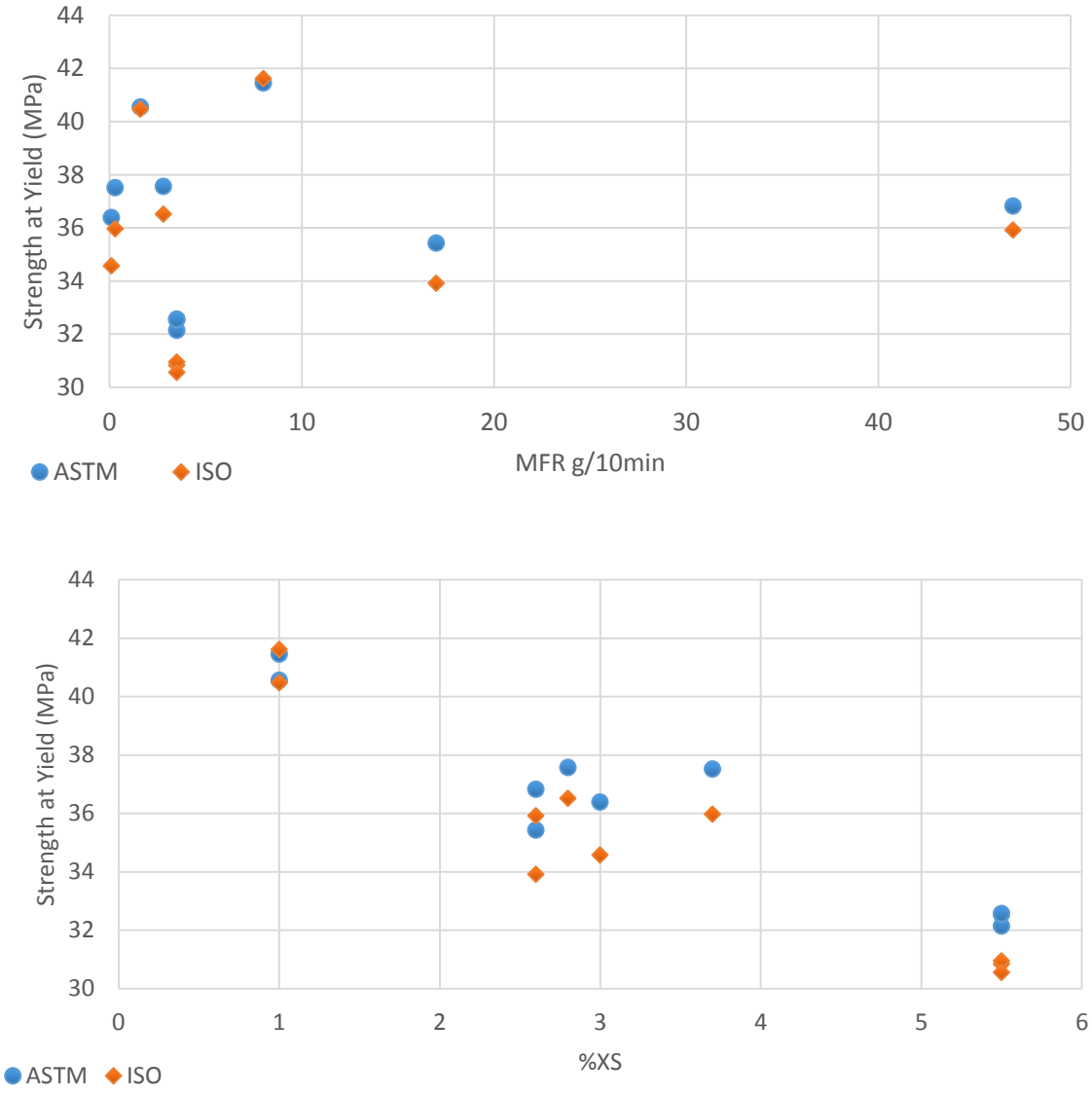

Figure 9: HPP tensile strength at yield as a function of MFR (top) and as a function of sample %XS (bottom).

The previous experiment on injection molding parameters found that tensile strength in a HPP sample does not depend on mold temperature or cycle time. This is in agreement with the ISO and ASTM comparison when uncertainty is considered. However, excluding samples with very low %XS, a slight increase in tensile strength at yield can be seen using the ASTM method. While it is within the uncertainty of the ISO value, this result was consistently found for seven samples. This may be due to a contribution from the differences in dimensions

between ISO and ASTM bars. In samples with low %XS this affect was negated by the dominance of the crystalline phase on the tensile properties.

Eleven ICP samples were also molded and tested following ISO and ASTM procedure for injection molding, Izod impact testing, flexural testing, and tensile testing. In addition, several of the samples were also Izod tested at 0°C and at -30°C, and Charpy impact tested at 23°C, 0°C, and -30°C.

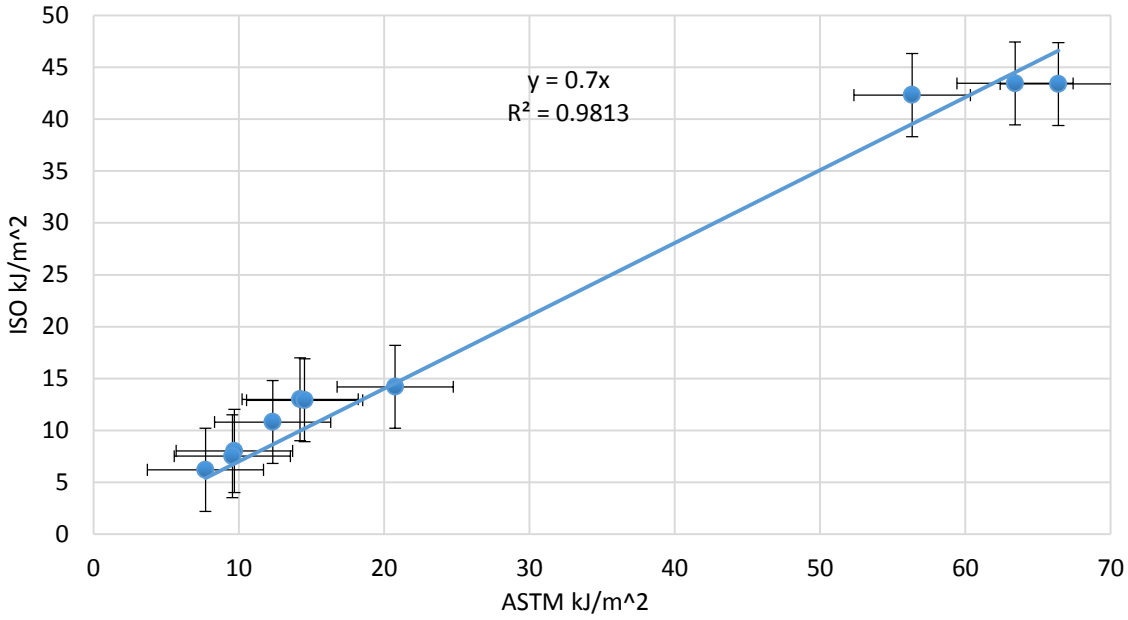

Figure 10: ICP Izod impact resistance ISO and ASTM comparison

For the Izod impact test, it was found that for ICP resins the ISO impact resistance values are approximately 0.7 times the ASTM values (Figure 10). This correlation was done using kJ/m² units, and may differ if the ASTM preferred J/m unit is used. The results for each method were compared as a function of MFR and Fc (Figure 11). The greatest difference between ISO and ASTM impact results occurs in samples with high Fc and was not related to sample MFR. Low and moderate Fc ICP samples did not show a significant difference in Izod values when the two methods were compared.

In the previous molding conditions experiment, it was found that cycle time and mold temperature did not affect the impact resistance of ICP samples. This is the same as the results for all ICP polymers tested except for the high Fc resins with the greatest impact resistance. The high Fc samples have a larger amorphous propylene-ethylene phase which leads to higher ductility and impact resistance. These samples have a higher Fc than the ICP 1 used in the previous set of experiments so it is possible the results from that are not consistent with high Fc very ductile samples. The dimensional differences between ISO and ASTM test specimens may also have contributed to the observed differences in impact resistance for ICP resins.

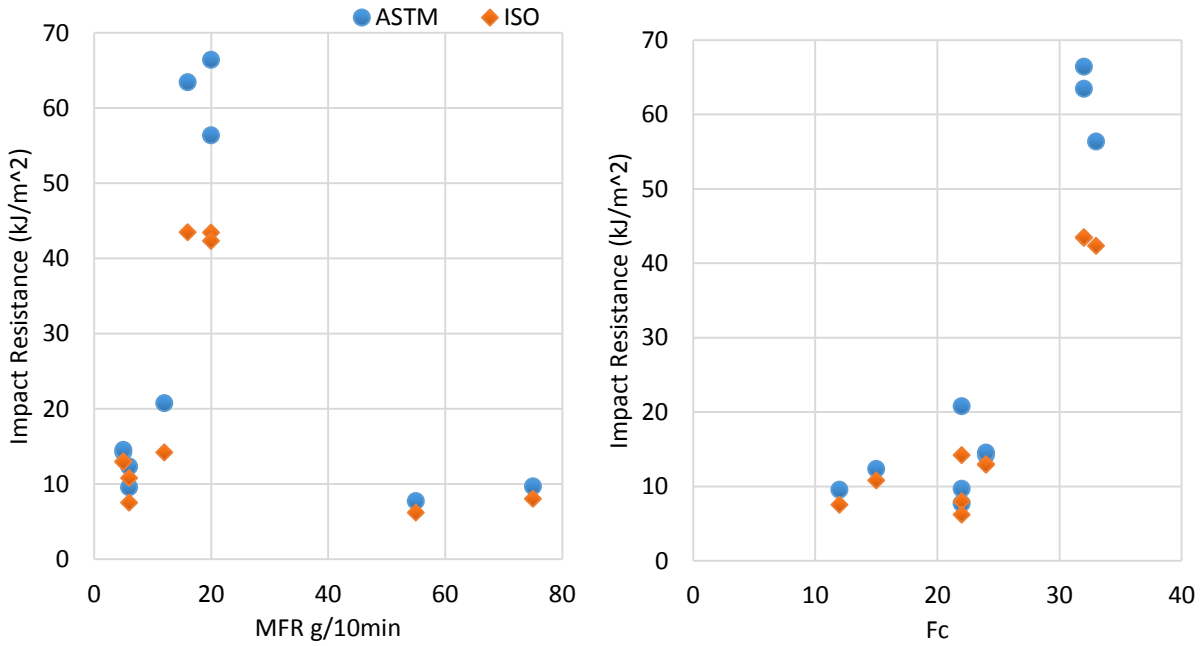

Figure 11: ICP Izod impact resistance as a function of sample MFR (left) and as a function of sample Fc (right).

Several of the ICP resin samples were subject to additional Izod impact testing at 0°C and -30°C (Figure 12). The difference in impact resistance shown in high Fc samples decreased as samples were cooled to lower temperatures. At 0°C and -30°C there is very little difference between the results reported with ISO and ASTM methods. This is expected as cold temperatures make samples more brittle and decrease the ductility of the amorphous regions.

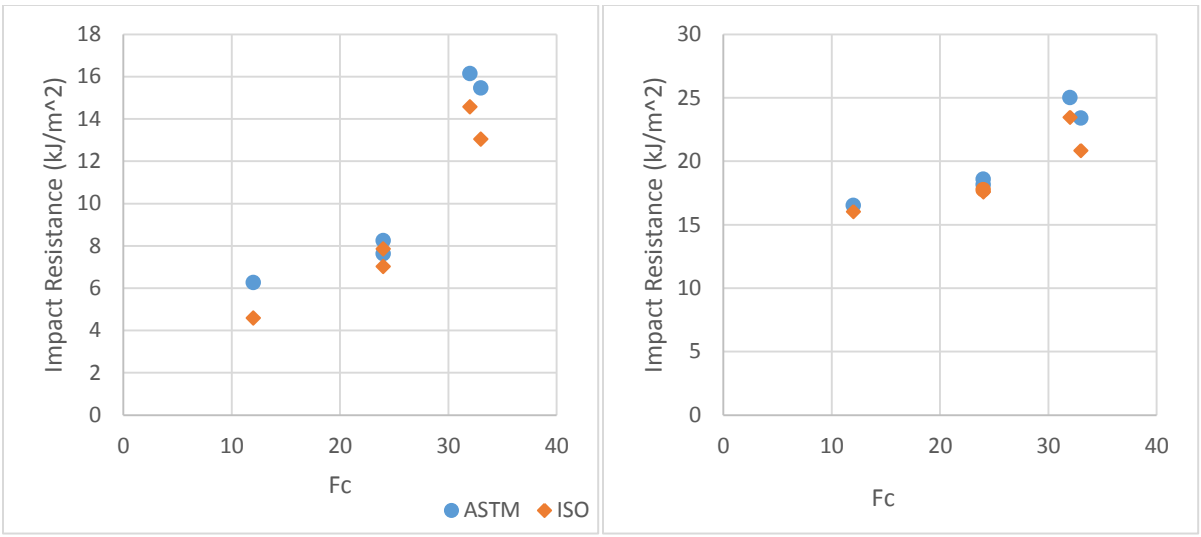

Figure 12: ICP Izod impact resistance (left) at 0°C and (right) at -30°C

The ICP samples were also flex tested and the ISO chord modulus results and ASTM secant modulus results were compared (Figure 13). There was no apparent difference between the results obtained using each method. The same is true for tensile strength at yield for the ICP samples. Based on the previous set of experiments, the ISO mold temperature contributed slightly to a lower flex modulus. However, this must be negated either by the differences in sample dimensions or modulus calculation between ISO and ASTM. The ICP tensile strength findings are in alignment with the results from the injection molding parameter experiments.

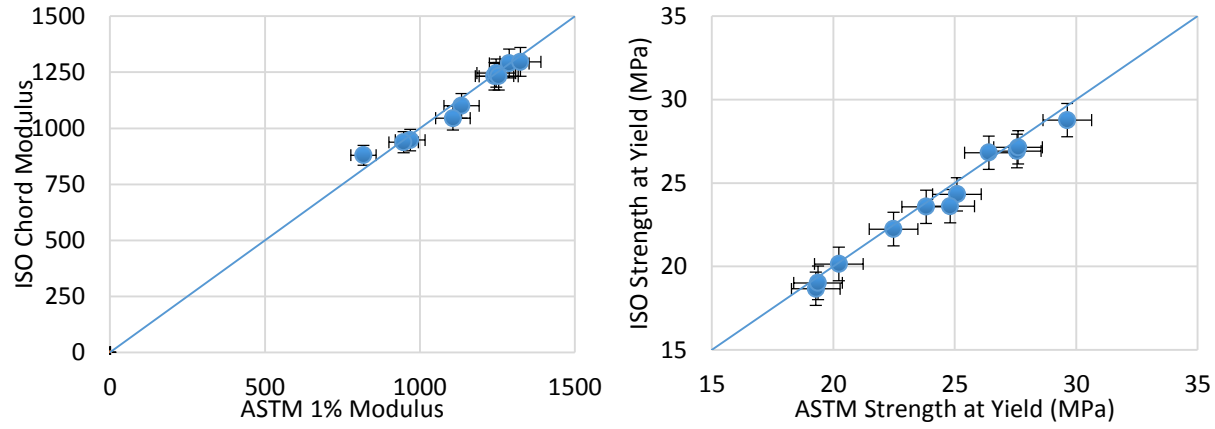

Figure 13: ISO and ASTM ICP flexural modulus comparison (left) and tensile strength at yield comparison (right).

A selection of the ICP samples used were also Charpy impact tested. The Charpy tests were done at 23°C, 0°C, and -30°C (Figure 14, Figure 15). At room temperature, the ICP Charpy impact resistance results show a similar pattern to the Izod impact results. In the resins with high Fc, Charpy testing using the ASTM method yielded higher impact resistance measurements than when using the ISO method. In resins with lower Fc, the increase in impact resistance with the ASTM method was observed to a lesser degree. As with the Izod impact test, the differences in sample dimensions between ISO and ASTM are likely the largest contributor to the measured differences in impact resistance.

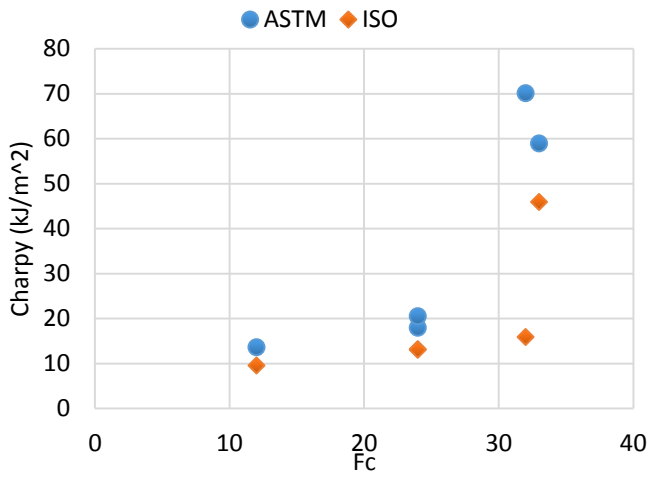

Figure 14: ICP 23°C Charpy testing

At lower temperatures the ICP resins become more brittle and impact resistance is decreased (Figure 15). This led to a smaller difference between results from ISO and ASTM methods when tested at -30°C. At 0°C, the results still showed a significant increase when measured following ASTM procedure. As it was previously found that injection molding conditions did not affect the impact resistance of ICP resins, this difference can be attributed to the greater depth of the ASTM test specimens.

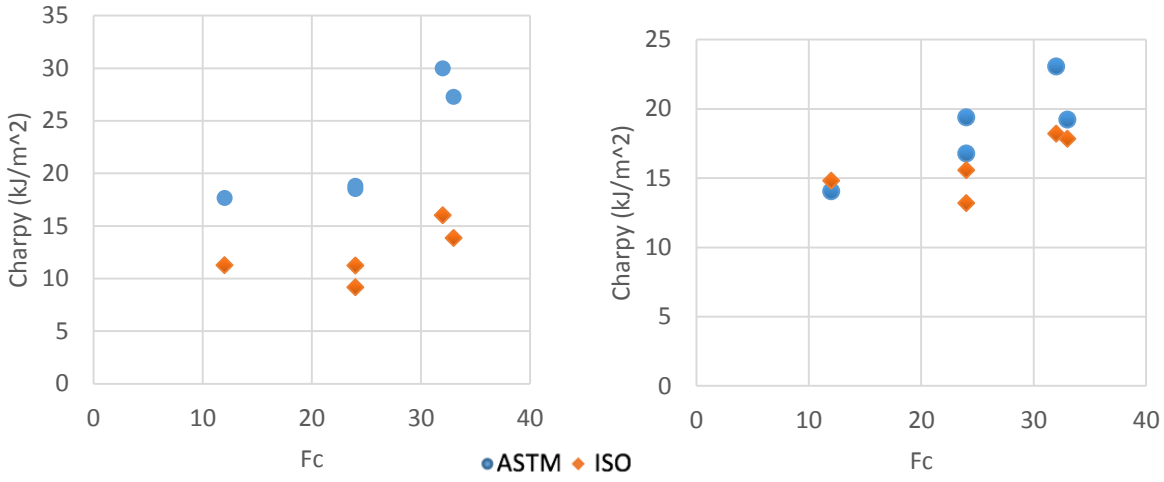

Figure 15: ICP Charpy impact resistance at 0°C (left) and at -30°C (right).

In addition, two RCP resin samples were tested using both ISO and ASTM procedures for molding and analytical testing. The Izod impact resistance, flexural modulus, and tensile strength at yield were measured for these two resin samples (Table 3).

Resin	MFR (g/10min)	Ec %	Izod				Flex Modulus		Tensile strength at Yield	
			ASTM		ISO		ASTM	ISO	ASTM	ISO
			(J/m)	(kJ/m^2)	(J/m)	(kJ/m^2)	(MPa)	(MPa)	(MPa)	(MPa)
RCP1	35	3	45	4	34	4	901	863	26	24
RCP2	7	6	167	17	80	10	614	580	21	19

Table 3: RCP resin ISO and ASTM testing results.

For these RCP resins, it was found that the ISO procedure reports a negligibly lower flexural modulus and tensile strength at yield than ASTM procedure. In the lower MFR resin, ISO procedure reported a lower impact resistance, and the other RCP resin showed the same impact resistance using both methods. As only two RCP samples were tested, it was difficult to establish correlations that apply to all RCP resins. ISO method values were about 5% lower for the flexural modulus and around 10% lower for tensile strength than ASTM method. Both of these differences are within the uncertainty range for the equipment used and can be

considered to be negligible. Overall, the two RCP samples followed a similar pattern to the ICP samples tested.

Optical Properties

In addition to mechanical properties, the differences due to ISO and ASTM methods in a resin's optical properties was also considered. Two clarified ICP samples were injection molded using eight different sets of conditions and the haze values were measured (Figure 16).

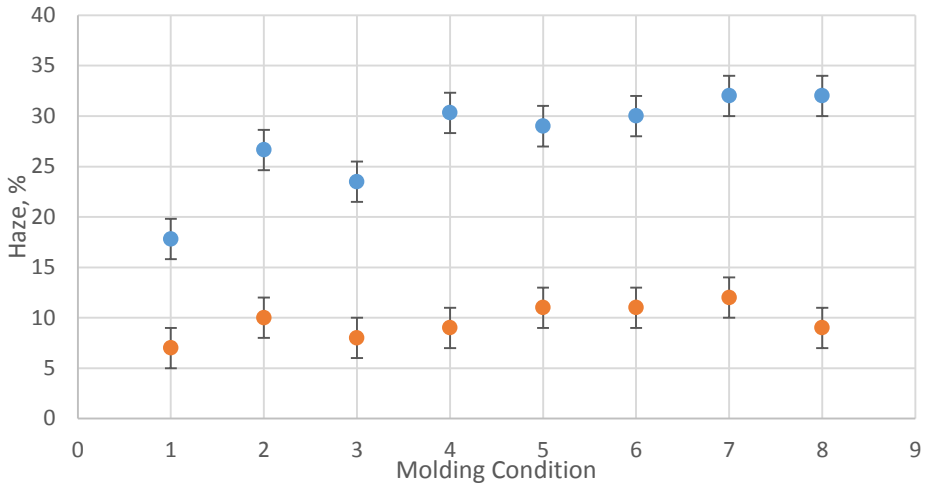

Figure 16: Optical haze of two clarified ICP resins processesed with various molding conditions.

The haze measurements for one of the resins were very low, and no distinction between the results from different molding conditions could be discerned. For the other sample it was clear that injection molding conditions one and three produced samples with lower haze values than the other molding conditions. Sets one and three were the only two molding parameter sets to use a mold temperature of 20°C, colder than the 45°C or 60°C used in the other molding runs. The other parameters considered (melt temperature, injection pressure, and injection velocity) produce no significant difference in sample haze. The colder mold temperature promotes faster cooling of the polymer melt when it is injected. This inhibits the formation of crystals and crystal size growth as the cooled chains are unable to move into spherulite

formation. Large crystal spherulites and a high overall crystallinity contribute to light scattering and haze in the sample, so a cold mold temperature was the most effective parameter in lowering haze in injection molded resin samples.

Modeling

Computer simulations were run to gain a better understanding of the injection molding process. The first simulations investigated the effect of mold temperature on flow induced residual stress and crystallinity (Figure 17). Mold temperatures of 40°C and 60°C were considered. From the simulation models, it is evident that mold temperature had a significant impact on the crystallinity of an injection molded sample. It also had an effect on the flow induced residual stress, a contributing factor from the melt flow on resulting chain orientation.

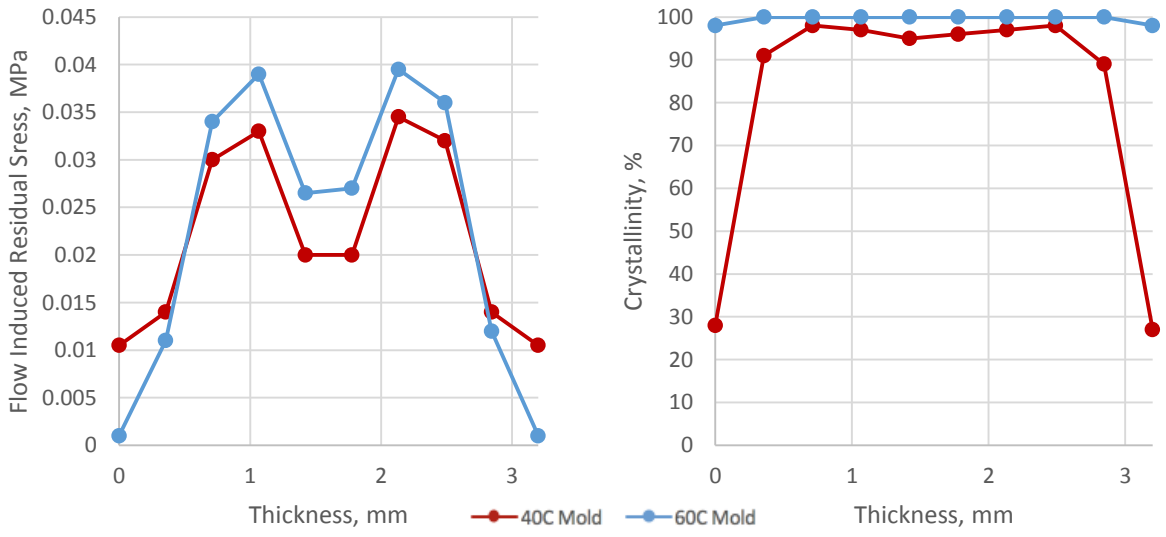

Figure 17: Computer simulated tensile bar mold flow induced stress (left) and relative crystallinity (right).

From this simulation it was apparent that colder mold temperatures produce a sample with lower overall crystallinity, and a more defined oriented skin layer at the sample edges. In the 60°C mold model, the edges of the bar were near to 100% relative crystallinity. In the 40°C mold model, the crystallinity dropped to around 30%in the skin layer. The colder mold

temperature model also showed a dip in crystallinity in the core region compared to the warmer mold. In addition, the colder mold led to higher stress at the sample edges, and lower residual stress in the sample core. This is because of the temperature dependence of polymer flow. As the melt cooled near the edges of the bar, shear stress was higher than in the model with a warmer mold. This supports the explanations given previously that the fast cooling in a colder mold contributed to overall lower crystallinity in the final injection molded sample.

The second set of simulations show the temperature profile across the thickness of ASTM and ISO bars 10 seconds after injection and at the end of the molding process (Figure 18). For ASTM, the end of the molding process is after 45 seconds, and for ISO it is after 60 seconds. The simulations shows that 10 seconds into the injection process ISO bars had a higher core temperature and a lower surface temperature. At the end of the molding process, the ISO bar had a lower temperature across the entire thickness. This simulation is evidence for the early explainations that the ISO method promotes a faster cooling rate in the sample than the ASTM method.

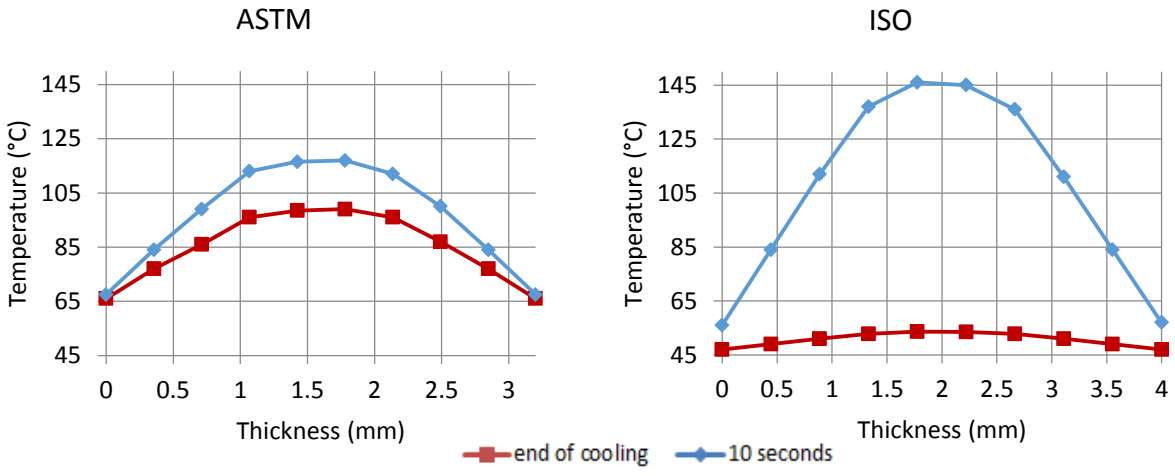

Figure 18: Computer simulated temperature profile across thickness of bar during injection molding

Another simulation was done to show the relative crystallinity during the molding process (Figure 19). In this simulation the core and skin layer morphology can cleary be seen during the molding process. At the end of cooling, the ASTM simulation showed a uniform crystallinity across the bar. The ISO simulation showed around 80% relative crystallinity on the sides of the bar in the skin region. In addition, the ISO bar showed a lower crystallinity than ASTM after ten seconds into the injeciton molding run. The results of this simulation agreee with the previous simulation into the temperature profile. As the ISO method promotes faster cooling of the polymer melt, the polymer chains become frozen faster and are unable to form as many crystals, especially in the skin region nearest to the mold face.

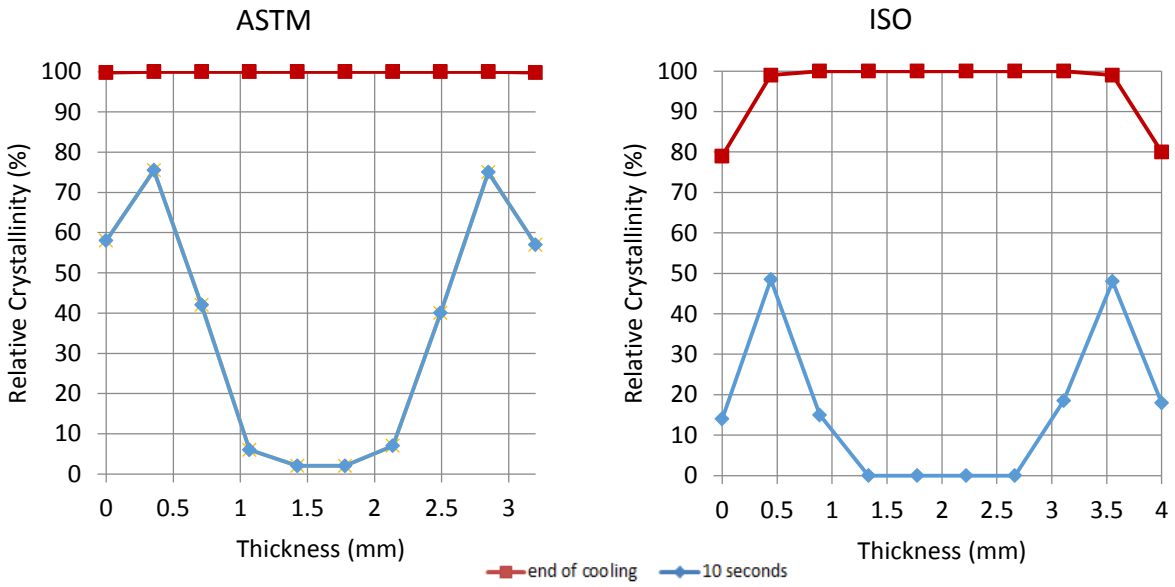

Figure 19: Computer simulated relative crystallinity across thickness of bar during injection molding

Further computer simulations were done on the relative crystallinity and volume shrinkage across the thickness of a 1mm step-chip mold at threee different temperatures (Figure 20). This simulation was useful to investigate the impact of mold temperature on the optical properties of a sample. It can be seen that mold temperature is proportional to relative

crystallinity and volume shrinkage. Volume shrinkage can be used a measure of the degree of orientation and alignment in the polymer chains. As oriented chains cool they shrink more than less oriented random chains. This is why the volume shrinkage shows peaks at the more highly oriented edges of the step-chip molds. This simulation provides further support for the optical properties results discussed earlier. Colder mold temperatures inhibit crystal formation and growth as shown in the simulations, which in turn leads to a sample with better optical properties including lower haze.

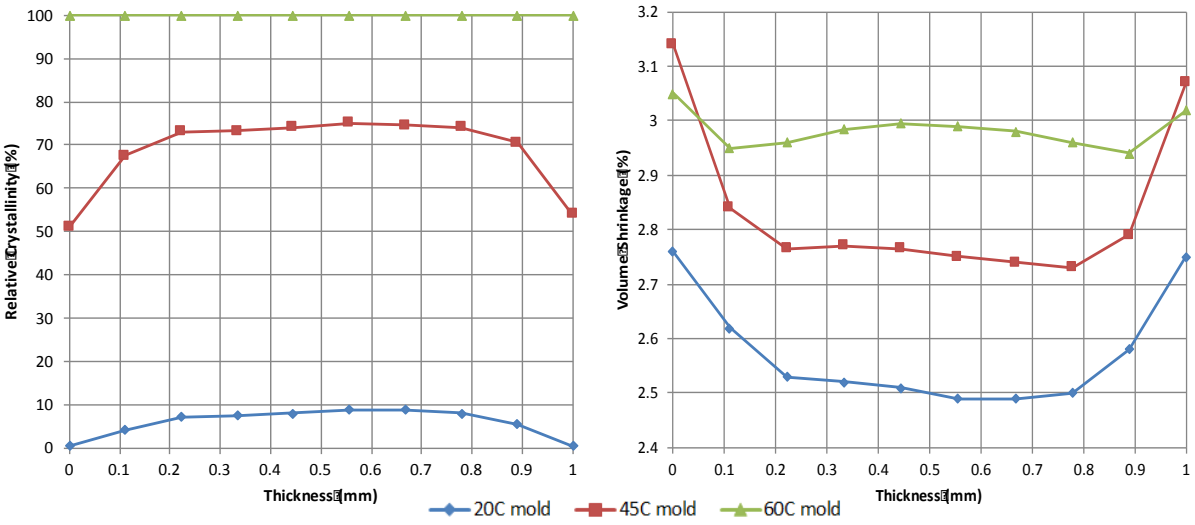

Figure 20: Computer simulated step-chip mold relative crystallinity (left) and volume shrinkage (right).

The findings from the computer simulations were in agreement with the experimental data from the injection molding parameter and the ISO and ASTM comparison studies. The simulations offer an explanation for many of the results observed during the experimental injection and testing. Through these models conditions during injection molding were able to be simulated that could not be easily measured experimentally.

CONCLUSION

This research project demonstrated that the many variables in the injection molding process can have an effect on the final mechanical properties of a molded polypropylene part. Both experimentally gathered data and computer simulations showed that the mold temperature and physical part dimensions are key parameters in the resulting mechanical properties of an injection molded part. Computer modeling showed that mold temperature plays an important role in crystal formation in a polymer melt, and experimentally mold temperatures were shown to have an impact on the impact resistance and the flexural modulus of some polypropylene samples. In addition, mold temperature was found to be the most important injection molding parameter in affecting the optical properties of a sample. Other molding conditions did not induce a significant change in sample haze measurements, but decreasing mold temperature was found to decrease the reported haze value. When directly comparing ISO and ASTM testing methods, the sample dimensions were also found to play a role in determining the final reported values for several mechanical properties.

While there are many differences between the polypropylene injection molding and analytical testing methods defined by ISO and ASTM specifications, the results obtained using each method are comparable for many of the major mechanical tests. For both the flexural modulus and the tensile strength at yield, ISO and ASTM methods can be expected to report the same result for a wide spectrum of polypropylene resin samples. However, there is variance in the impact resistance reported using each method for several resin types. In HPP samples with low MFR (<1 g/10min.) it was found that that ISO procures result in an Izod impact resistance value up to 50% greater than the result found using ASTM procedures. For ductile ICP samples with high Fc, it was found that the ASTM method reports an Izod value up to about 50% greater

than the value from the ISO method. When the impact resistance tests were conducted at lower temperature, the differences between the two methods were minimized. These finding have been largely explained through the experiments done on injection molding parameters and the results from the computer simulations. The results of this report are very useful for comparisons between ISO and ASTM performance values for polypropylene and provide some understanding of the important factors during the injection molding and analytical testing processes.

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Biography

Henry J. Sokol was born in 1994 in Seattle, Washington. He is the son of Jim and Deborah Sokol and has an older brother, Miles, and a younger sister, Emily. Henry graduated from Inglemoor High School in Kenmore, Washington in 2013. He did his undergraduate work at Johns Hopkins University where he studied Chemical and Biomolecular Engineering, earning a Bachelor of Science degree in 2017. Following that he entered into the Master of Science in Engineering degree program in the same department. As part of his graduate studies, Henry worked in a co-op position in the Specialty Catalysts R&D group at W.R. Grace & Co. in Columbia, Maryland. The work for this thesis is a result of a project he did while at W.R. Grace.